Loss on ignition

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by

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Preface

This guide deals with determining loss on ignition on soil sorts.

The guide is part of a series, which explain the execution of geotechnical classification experiments as carried out at the Geotechnical Engineering Laboratory at Aalborg University.

The guide is constructed as follows:

- *Appertaining standards*
- *Definitions*
- *Apparatus*
- *Equipment calibration*
- *Preparing the test sample*
- *Procedure for experiment*
- *Calculations*
- *Reporting*
- *Remarks*
- *Schema for experiment execution*
- *Appendix, if any*

It is recommended that the user of this guide reads the entire guide before the experiment is started.

Numbering of figures in the text is indicated by { }.

Units are indicated by [ ], e.g. [%].

Appertaining standards

The experiment is based on and further described in the standards DS 204 and DS/EN 1997-2, Eurocode 7: Geoteknik – Del 2, Nationalt Anneks N.

Definition

Ignition loss is used to determine organic material in non-cohesive soil.

Dry matter: Material after drying at 50^{o}C.

Ignition remnant: Material after annealing at 550^{o}C.

Loss on ignition: The difference between dry matter and ignition remnant in percentage.

Apparatus

Apparatus used in the experiment. Numbers refer to figure 1.

- Annealing furnace {1}
- Annealing tray {2}
- Crucible {3}
- Crucible tongs {4}
- Mortar
- Drying oven at 50^{0}C
- Desiccator
- Scale, weight accuracy 0.1 mg = 0.0001g

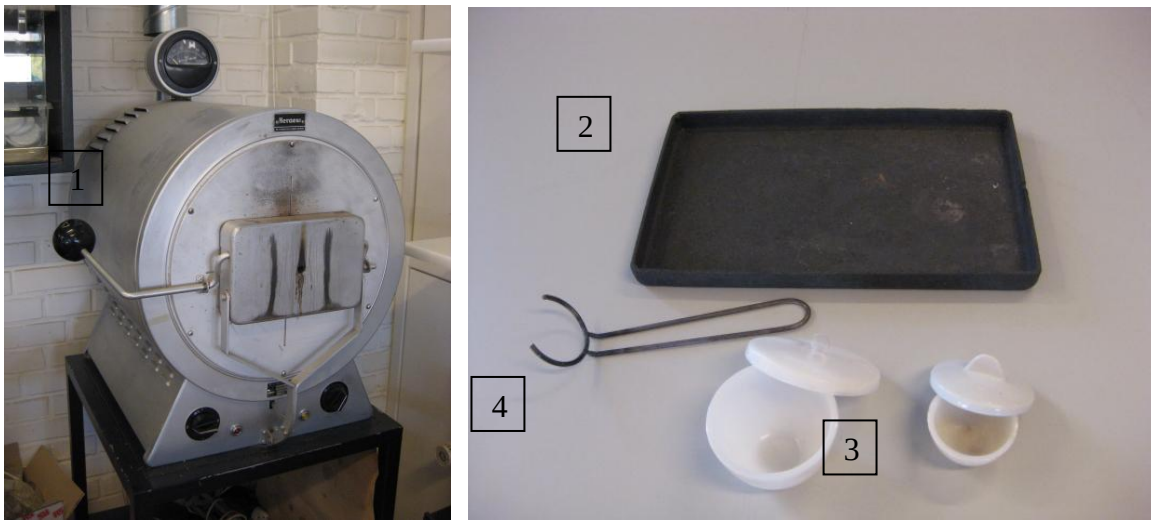


Figure 1: Apparatus for loss on ignition experiment. Numbers refer to apparatus listed above.

Equipment calibration

The equipment does not require any calibration prior to the experiment. The annealing furnace temperature must, however, be checked annually.

The crucibles are annealed twice annually. The crucibles are checked and discarded if any chips or the like are found.

Preparing the test sample

The test sample and the crucibles must be dry before annealing so that no interstitial water is found in the sample of moisture on the crucible. The sample and the crucibles are prepared the day before and placed in the desiccator on the day of annealing.

- A suitable subsample on 100-200 g is taken and placed in steel or ceramic bowl.
- The sample and the crucibles with lid are dried at 50^{o}C over night or until the weight changes to less than 0.1 % at an additional 1 hour drying.
- The samples and crucibles are placed in the desiccator after drying.

Procedure for the experiment

- Check whether the annealing furnace is clean
- The sample is taken from the desiccator.
- The sample is grinded down in a mortar, figure 2.
- Crucible with lid is taken from the desiccator and the number under the bottom is noted down.
    - If the number is not there or clear, one is written with a pencil. Extra lead is removed with a fine brush.
- The crucible with lid is weighed (Dig) with a weight accuracy of 0.1 mg, figure 3. The scale box around the scale must be closed when the scale is being read.
    - Note that the analysis scale with a weight accuracy of 0.1 mg must not be loaded by more than 220 g.
- 20-25 g of the fine, grinded material is placed in the crucible. Do not do this while the crucible is on the scale.
- The crucible with lid and sample is weighed ($Dig+W_s$) with a weight accuracy of 0.1 mg.
- The crucible is placed on the annealing tray, and the placement is marked on a drawing of the annealing tray so it will be possible to recognize the crucible if the number should disappear during annealing.

Figure 2: The dried matter is grinded in a mortar.

Figure 3: Crucible with lid on the scale. The sides of the scale box are closed.

- The annealing tray is placed in the cold annealing furnace, figure 4, which is closed. The annealing tray is placed so the small groove in the one end is facing towards the opening of the annealing furnace.
- Make sure that the vent control handle on the backside of the furnace is open, figure 5.
- The annealing furnace is turned on to 550^{o}C.
- When the temperature in the annealing furnace reaches 550^{o}C, the time is noted down, and an additional 4 hours of annealing is done.

Figure 4: The annealing tray with crucibles placed halfway into the cold furnace.

Figure 5: The open vent control handle.

- After annealing, check whether the whole sample is done annealing. If necessary, rake lightly in the crucible with a piece of steel wire. The sample is done annealing with the sample has the same colour down through the entire sample.
    - If the sample is not done annealing, it is annealed for one additional hour after which the sample is checked again.
- When the samples are done annealing, the annealing furnace is shut off, and the annealing tray is pulled halfway out of the furnace, and the crucibles are taken with crucible tongs and placed on a tray, figure 6.
    - Do not attempt to remove the annealing tray as it is too hot and heavy for it to be moved without first being cooled down.
- The crucible with the annealed sample is placed gently with the annealing tongs in the vacuum desiccator where it must be for at least 1 hour or until it has room temperature, figure 7. It is important that the lid on the crucible is placed correctly as the sample can whirl up otherwise when the vacuum desiccator is turned off.
- The cooled crucible with lid is weighed immediately after it has been removed from the desiccator ($Dig+W_{gl}$).

The annealing furnace must not be closed before it has cooled down to approx. room temperature.

Figure 6: The crucibles are removed with annealing tongs. If necessary, use gloves as it is very hot by the open furnace.

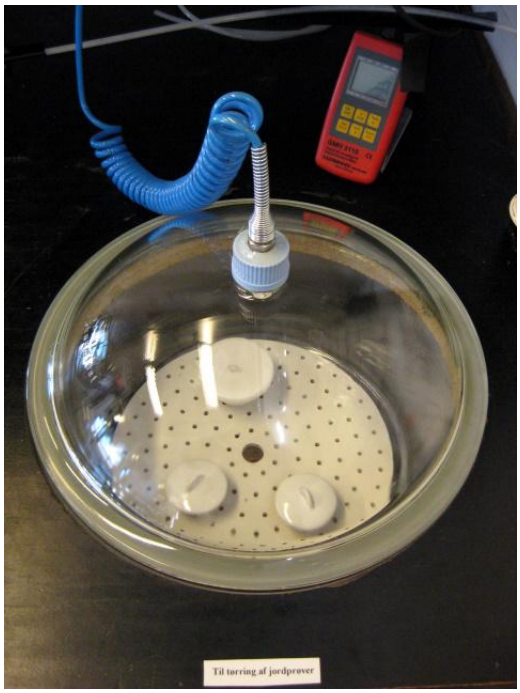


Figure 7: Crucibles for cooling down in the desiccator.

Calculations

The loss on ignition is given by the weight loss as a result of the annealing over the weight of the dry matter.

$$GL(\%) = \frac{weight\ loss}{W_s} = \frac{(Dig + W_s) - (Dig + W_{gl})}{(Dig + W_s) - Dig}$$

Reporting

The result is reported with an accuracy of 0.1 %.

Also, at which temperature the drying and annealing was done as well as any extra annealing time is noted.

Remarks

On the annealing furnace, the red lamp will be on when the annealing furnace is turned on. The yellow lamp will be on when the annealing furnace is heating up.

The annealing tray and crucibles must at maximum fill 50 % of the annealing space.

The crucibles must stand on an annealing resistant tray during annealing.

Before the annealing furnace is turned on, check whether the annealing furnace and tray are clean. Dirt or soil on the bricks in the annealing space can damage the annealing furnace.

If necessary, control of the annealing can be done on a separate crucible with the same amount and type of material. The separate crucible must be treated in the same manner and annealed in the same time as the crucibles which are being measured. In this manner, material will not be wasted when stirring in the crucible.

AALBORG UNIVERSITET

Loss on ignition

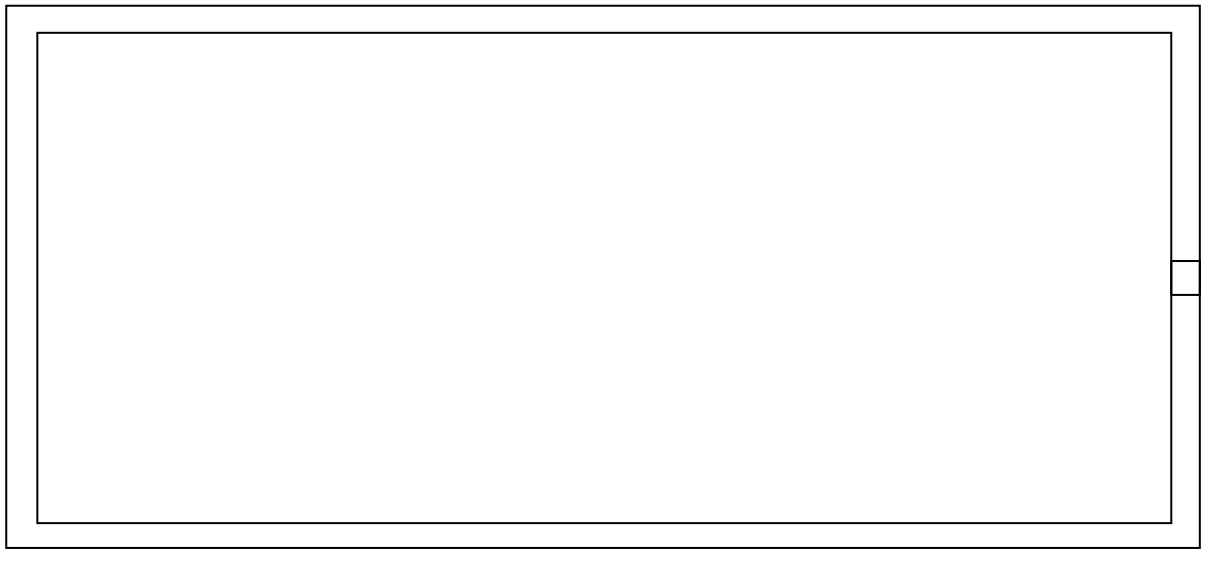
Case			Case no.
Examined on	to	Lab. no.	Borling no.
Controlled. d.	Approved d.	Level	Appendix no.

LOSS ON IGNITION

Sample	no						
Bowl	no						
Sample in drying cab.	h.						
Sample out drying cab.	h.						
Crucible	no						
Crucible W_{dig}	g						
Crucible + dry matter W_s	g						
Crucible in annealing furnace	h.						
Annealing furnace hot	h.						
Crucible out annealing furnace	h.						
Cold crucible + ignition remnant W_{gl}	g						
Weight loss	g						
Loss on ignition $GL(\%) = \frac{weight\ loss}{W_s} \cdot 100\%$	%						

ANNEALING TRAY

Placement of crucibles are marked with circles and numbers.



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